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ON THE STRENGTH OF SINTERED SILICON CARBIDE

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16. Abstract The effects of surface treatment and microstructure, esp. abnormal grain growth, on the strength of sintered SiC were studied. The surfaces of sintered SiC were treated with 400, 800 and 1200 grit diamond wheels. Grain growth was induced by increasing the sintering times at 2050°. The beta to alpha transformation occurred during the sintering of beta-phase starting materials and was often accompanied by abnormal grain growth. The overall strength distributions were established using Weibull statistics. The strength of the sintered SiC is limited by extrinsic surface flaws in normal-sintered specimens. The finer the surface finish and grain size, the higher the strength. But the strength of abnormal sintering specimens is limited by the abnormally-grown large tabular grains. The Weibull modulus increases with decreasing grain size and decreasing grit size for grinding.					
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THE EFFECTS OF SURFACE FINISH AND GRAIN SIZE ON THE STRENGTH OF SINTERED SiC

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I. Introduction

During the last decade, there have been many studies on the development and usefulness of the ceramics industry, especially on ceramics engineering. Generally, the raw material for ceramics manufacture was recognized as SiC (silicon carbide) and SiN (silicon nitride), and the study is naturally concentrated on the development of both materials. Because these materials are recognized as superior in their characteristics of heat-resistance, wear-resistance, and others to metal raw materials, they are greatly appreciated as proper materials for industrial ceramic uses. SiC was investigated by Prochazka [1] as a sintering effect of carbon and boron, and thereafter research on the effects of the sintering function of SiC accelerated, mainly focusing on its mechanical clearance [2,3,4].

Nevertheless, SiC was not used as the raw material for engine construction despite its superior characteristics compared with those of metal, because of the corrosive characteristics of carbon compounds. Therefore, it was necessary to show various data such as K_I (stress intensity factor), the K_I -V (crack velocity) diagram, m (Weibull modulus) [5], the SPT (strength-probability-time) diagram, besides its strength intensity. This research has focused on investigating the relationship between m and K_I using SiC which has a theoretical density of 96% or greater. Also, research was done to observe the influence of the microstructure of materials on m and K_I in the final stage.

Further, surface treatment of SiC was administered in step 5 and the influence of surface treatment results on the m value were investigated. Therefore, as a result, this research has focused on

the influence of m value increase on the microstructure and surface treatment of SiC.

II Experimental method

2-1 Raw materials

The raw materials for the synthesis of SiC are ultra-fine powder of SiC (betarundum) of purity more than 99% and boron and carbon which are used for the acceleration of sintering. The SiC powder has a β -SiC crystal structure and the median diameter of the powder is 0.26 μm and the powder particles are spherical. Carbon 2 w/o and boron 1 w/o are added to the SiC powder mix for 24 hours with acetone in a WC ball-mill then dried and used for the sintering process.

2-2 Formation and Sintering

The primary formation was done in a 0.8 x 3.5 cm steel die under a pressure of 250 kg/cm² and then isostatic pressing was done under 1930 kg/cm² pressure. At this point, the formation density gained 56-58% of the theoretical density. Sintering was done under 2050° C, atmosphere Ar for 30 minutes and then extended sintering was done for 1-1.5 hrs for the formation of particle sintering.

2-3 Strength experiment

We measured 4-point strength degree using a bar of material of size of 0.6 x 0.4 x 2.85 cm. The inner span was 0.8 cm and the outer span was 2.4 cm. The experimental bar was sintered and four different types were prepared: one had an untreated surface and the others were treated with diamond wheels of 400 grit, 800 grit, and 1200 each. We polished the bar longitudinally, with a mark on the corner of the bar. To see the blunting effects, the sintered bar of material was polished with a 400 grit diamond wheel and heat treated at 2,000° C Ar atmosphere for 20 minutes, then the strength intensity was tested.

2-4 Toughness Test

The toughness of sintered SiC was determined by the impact technique (Vicker's impactor, 136 degrees) after polishing with 0.25 μm diamond paste. The equation used for the determination of toughness and hardness was as follows:

$$K_{Ic} = 0.0726 \left(\frac{P}{C^{3/2}} \right) \text{-----(1)}$$

$$H = \frac{P}{2 a^2} \text{-----(2)}$$

Here, P = applied load

C = Half of crack length

a = Half of the impactor cross-section

Lawn and Marshall [7] compared the character of ceramic materials using toughness and hardness at the same time. They defined it as brittleness and expressed it as follows:

$$B = \frac{H}{K_{Ic}} \text{-----(3)}$$

2-5 Observation of microstructure and X-ray analysis

To observe the microstructure of the sintered SiC, the sintered SiC specimen was polished with a 1200 grit diamond wheel and further polished with 6 μm , 1 μm , 0.25 μm fine diamond paste. Etching was done with boiling Murakami solution for the β -SiC particles and with a KOH, KNO_3 mixture solution for the α -SiC particles. The etched specimen of sintered SiC was observed under electron microscopy. The crystal structure of the sintered SiC was revealed under X-ray analysis using a $\text{CuK}\alpha$ ray.

III. Experimental results and evaluation

3-1 Sintering

SiC powder which had added boron and carbon as the accelerator

was sintered for 30 minutes in an Ar atmosphere of $2,050^{\circ}\text{C}$, and its length shrank 16% and density more than 96%. The average particle diameter was 2-3 μm by point counting, and there were partial abnormal α -SiC particles. This is illustrated in Figure 1a. Figure 1b had an extended sintering time of 1.5 hours at 2050°C and its fine structure is illustrated. In this growing particle specimen, abnormal growth of particles occurred, and the largest α -SiC particle was measured at 150 μm . The abnormal particles are illustrated in Fig.2. The clearest appearance in the specimen with extended sintering time was the movement on α -SiC. α -SiC connects the particles of β -SiC around them and extends growth in the longitudinal direction, increasing the aspect ratio. This phenomenon particularly occurs in the α -SiC which is in a better condition for growth or modification. Table 1 shows the microstructure which changes according to the sintering time.

3-2 Bending Strength

Figure 3 shows the results of investigation of the influence on strength according to the surface treatment. In this figure, the sintered specimen illustrates a strength increase of 40% when treated by the 1200 grit diamond wheel compared to the untreated specimen. When treated by 400 grit, a 27% increase of strength was shown. This phenomenon shows that bending strength is greatly influenced by the surface condition. In Fig.3, there was no influence on surface treatment above 400 grit. This phenomenon explains the fact that surface treatment has influenced the remaining cohesive force of the particles or breaking down of the particles.

Figure 4 illustrates the relationship between the strength of the sintered specimen and the surface treatment. Even though the surface treated specimen increases in strength compared to the untreated specimen, the surface structure becomes fine, like the

half-hour sintered specimen, so that it can be said generally not to increase in strength. Considering the fact that in Table 1 the density is almost same, the strength seems greatly influenced by the abnormal particles. Considering the specification of the MOR strength test, it can be said that great conversion of strength occurs according to the position of abnormal particles in the specimen which was observed by the 4-point bending. This result shows the fact that the strength connection which restricts the strength is not the same as for the 0.5 hr sintered specimen. This is also the reason that the value of the Weibull modulus(m) drops in Fig.7. In Fig.4, it can be seen that abnormal growth of particles is more frequent in a specimen where micropolishing was done and the breaking strength is decreasing. This is because the abnormal particles are a major function for the cohesive force, and as we can see in Fig.1, the average particles grow as obstacles per area compared with the 0.5 hr standard specimen. In the specimen where abnormal growth has happened, a slight breakdown of particles was seen.

3-3 Toughness Test

Figure 5 shows the difference in toughness and brittleness, and hardness of SiC sintered according to the length of time. The hardness shows the degree of resistance to material deformation, and the toughness shows the degree of resistance to the breakdown of the material. For the mechanical characteristics, hardness and brittleness increase but toughness decreases. This is because as the sintering time is extended, the average particle size grows and there is a strong relationship with the microstructure of SiC. Therefore, abnormal particles exhibit more sensitive responses to the breakdown stimulus and reliability decreases with abnormal particles compared with normal particles. This coincides with the fact of the experiment in Fig.7, where the Weibull modulus value m decreases.

3-4 Weibull Analysis

Figure 6 shows the distribution pattern of bending strength. The inclination was the Weibull modulus, m and more than 26 specimens were used. The inclination m was measured by applying the LS(least

square) method. It was interpreted that the increase of the Weibull modulus in the 400 grit polished specimen compared to the untreated one is the result of increased uniformity. Also the reason why the 1200 grit polished specimen has more Weibull modulus compared to the 400 grit polished one can be explained by the fact that diamond grit size influences the surface deficit. Koepke et.al.[11] defined the relationship as follows:

$$C_s = \alpha g_o (0.2 < \alpha < 1) \text{-----(4)}$$

where, C_s = surface deficit on the surface of materials
 g_o = grit size

Here, the value of α approaches 1 as the grit size becomes finer.

Figure 7 shows the comparison of bending strength after polishing the 0.5 hr sintered specimen with a 1200 grit diamond wheel for the purpose of finding the effects of fine structure of the sintered SiC on the Weibull modulus. The 1.5 hr sintered specimen showed normal distribution of the strength. The following can explain the reason for the low m value of the abnormal specimen compared to the normal specimen. There could be a variation of distribution of the strength, as the abnormal one restricts the strength.

As we can see in Fig.5, the brittleness increases in the specimen which has the abnormal particles, therefore, the breakdown rate of this specimen also increases accordingly.

IV. Conclusion

1. SiC has a spherical format when sintered at 2050° C for 30 minutes, and the size of the particles is 3-5 μm almost uniformly. When this specimen is sintered with a 6 μm diamond wheel, a strength increase can be visible up to 40%. This fact explains that the surface deficit of SiC would cause the strength to vary.

2. Abnormal particles grow when SiC has been sintered for 1.5 hr,

and the abnormal particle has a larger aspect ratio. The size reaches 150 μm .

3. The abnormal particle specimen exhibits less influence of surface treatment on strength. This is because the abnormal particles are a more important cause for strength variation.

4. The abnormal specimen showed an increase of strength from 14 GNm^2 to 21.4 GNm^2 , a decrease of toughness from 3.5 $\text{MNm}^{-3/2}$ to 2.6 $\text{MNm}^{-3/2}$. This results in an increase of the brittleness and increase in the breakdown rate of the sintered SiC.

5. Table 2 shows the Weibull modulus of a normal and abnormal sintered specimen ($\sigma_u=0$). Therefore, to use SiC for ceramic material it is necessary to protect from fine surface treatment and abnormal particle formation and to assist in increasing the Weibull modulus and breakdown strength.

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Table 1. Properties of specimens at various sintering times

property sintering time	average grain (largest grain size) size	microstructure	linear shrinkage (%)	sintered density (% T. D.)
0.5 hr	2-4 μm (12 μm)	spherical shape and uniform grain size	16.0	97
1.0 hr	8-10 μm (65 μm)	transient	16.1	98
1.5 hr	12-15 μm (150 μm)	large aspect ratio and exaggerated grain growth	16.2	98

Table 2. Summary of Weibull modulus for various materials

Hour	Surface Finish	m	σ_0
0.5 hr	As Received	5.14	1356
	400 Grit Diamond	5.99	3472
	800 Grit Diamond	7.15	2187
1.5 hr	1200 Grit Diamond	5.30	1766



(a)



(b)

Fig. 1 Microstructures of sintered SiC(2000 \times). sintering conditions: (a) 2050°C, 0.5hr, (b) 2050°C, 1.5hr



Fig. 2 Typical microstructure of abnormal grain growth (1000 \times). sintering conditions: 2050°C, 1.5hr

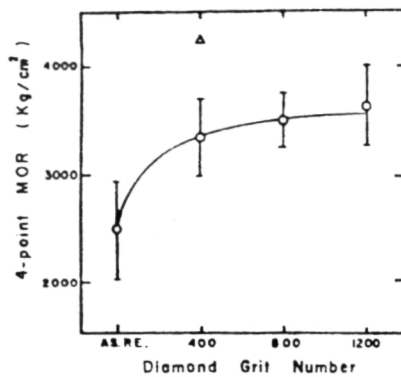


Fig. 3 MOR as a function of surface finish in sintered SiC. Above mark (Δ) indicated the strength of specimens which were annealed at 2000°C after surface treatment with 400 grit diamond. (AS. RE.=as received)

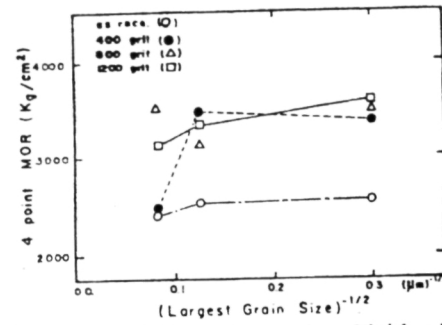


Fig. 4 Strength-Grain Size-Surface Finish relations in sintered SiC. (as rece.=as received)

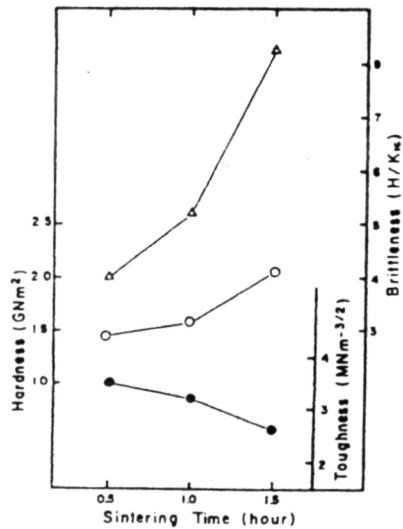


Fig. 5 Microhardness(○), toughness(●) and brittleness (Δ) as function of sintering times in sintered silicon carbide.